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UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 C.F.R. § 1.53(b))

Attorney Docket No. 602-1479

First Inventor or Application Identifier Cole

Title Evaporation of Liquids

Express Mail Label No. EL 388 803 025 US

PTO/SB/05 (4/98)

APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents.

 ADDRESS TO: Assistant Commissioner for Patents
 Box Patent Application
 Washington, DC 20231

1. ☒ Fee Transmittal Form (e.g., PTO/SB/17)
 (Submit an original and a duplicate for fee processing)
2. ☒ Specification [Total Pages 11]
 (preferred arrangement set forth below)
- Descriptive title of the invention
 - Cross References to Related Applications
 - Statement Regarding Fed sponsored R & D
 - Reference to Microfiche Appendix
 - Background of the invention
 - Brief Summary of the invention
 - Brief Description of the Drawings (if filed)
 - Detailed Description
 - Claim(s)
3. ☒ Drawing(s) (35 U.S.C. 113) [Total Sheets 3]
4. Oath or Declaration [Total Pages 3]
- a. ☒ Newly executed (original or copy)
- b. ☐ Copy from a prior application (37 C.F.R. § 1.63(d))
 (for continuation/divisional with Box 16 completed)
- i. ☐ DELETION OF INVENTOR(S)
 Signed statement attached deleting inventor(s) named in the prior application, see 37 C.F.R. §§ 1.63(d)(2) and 1.33(b).

5. ☐ Microfiche Computer Program (Appendix)
6. Nucleotide and/or Amino Acid Sequence Submission
 (if applicable, all necessary)
- a. ☐ Computer Readable Copy
- b. ☐ Paper Copy (identical to computer copy)
- c. ☐ Statement verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

7. ☐ Assignment Papers (cover sheet & document(s))
8. ☐ 37 C.F.R. § 3.73(b) Statement of Attorney
 (when there is an assignee) ☐ Power of Attorney
9. ☐ English Translation Document (if applicable)
10. ☐ Information Disclosure Statement (IDS)/PTO-1449 ☐ Copies of IDS Citations
11. ☒ Preliminary Amendment
12. ☒ Return Receipt Postcard (MPEP 503)
 (Should be specifically itemized)
13. ☐ Small Entity Statement(s) ☐ Statement filed in prior application, Status still proper and desired (PTO/SB-09-12)
14. ☐ Certified Copy of Priority Document(s) (if foreign priority is claimed)
15. ☐ Other:

* NOTE FOR ITEMS 1 & 13: IN ORDER TO BE ENTITLED TO PAY SMALL ENTITY FEES, A SMALL ENTITY STATEMENT IS REQUIRED (37 C.F.R. § 1.27), EXCEPT IF ONE FILED IN A PRIOR APPLICATION IS RELIED UPON (37 C.F.R. § 1.28).

16. If a CONTINUING APPLICATION, check appropriate box, and supply the requisite information below and in a preliminary amendment.

☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application No. _____ /

Prior application information: Examiner _____ Group / Art Unit: _____

For CONTINUATION or DIVISIONAL APPS only: The entire disclosure of the prior application, from which an oath or declaration is supplied under Box 4b, is considered a part of the disclosure of the accompanying continuation or divisional application and is hereby incorporated by reference. The incorporation can only be relied upon when a portion has been inadvertently omitted from the submitted application parts.

17. CORRESPONDENCE ADDRESS

☐ Customer Number or Bar Code Label (Insert Customer No. or Attach bar code label here) or ☒ Correspondence address below

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Signature	<i>William M. Lee, Jr.</i>	Date	4/17/00

Burden Hour Statement: This form is estimated to take 0.2 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Box Patent Application, Washington, DC 20231.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE THE APPLICATION OF)
Michael Cole)
SERIAL NO.: To be Assigned)
FILED: Herewith)
FOR: Evaporation of Liquids)

AMENDMENT ACCOMPANYING APPLICATION

Honorable Commissioner of
Patents and Trademarks
Washington, D.C. 20231

Dear Sir:

Before calculation of the filing fee in the United States for this application, it is requested
that the application be amended as follows:

In the Claims

Claim 3, line 1, delete "or claim 2"

Claim 4, line 1, delete "any one of claims 1 to 3" and substitute - - claim 1 - -

Claim 5, line 1, delete "any one preceding" and after "claim" insert - - 2 - -

Claim 6, line 1, delete "as dependent upon claim 2"

Claim 7, line 1, delete "or claim 6"

Claim 8, line 1, delete "any one of claims 5 to 7" and substitute - - claim 5 - -

Claim 12, line 1, delete "any one of claims 9 to 11" and substitute - - claim 9 - -

Claim 13, line 1, delete "any one of claims 9 to 12" and substitute - - claim 9 - -

Claim 14, line 1, delete "any one of claims 9 to 13" and substitute - - claim 9 - -

Claim 15, line 1, delete "any one of claims 9 to 14" and substitute - - claim 9 - -

Claim 16, line 1, delete "any one of claims 5 to 15" and substitute - - claim 5 - -

Claim 17, line 1, delete "any one of claims 5 to 16" and substitute - - claim 5 - -

Claims 18 and 19, line 1, delete "any one of claims 9 to 15" and substitute - - claim 9 - -

Cancel claim 21 without prejudice as not being in accordance with U.S. practice.

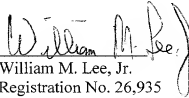
Remarks

The above amendments are being made in order to eliminate multiple dependency and improper multiple dependency, as well as an improper claim, before calculation of the application filing fee.

Examination of the application on its merits is awaited.

April 17, 2000

Respectfully submitted,


William M. Lee, Jr.
Registration No. 26,935

Lee, Mann, Smith, McWilliams,
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(312) 368-6620
(312) 368-0034 (fax)

0550693-041700

Please acknowledge
receipt of the
enclosed:
Michael Cole

Evaporation of Liquids

SERIAL NO: To be assigned

FILING DATE: Herewith

1. Utility Patent Application Transmittal
2. Fee Transmittal for FY 1999 in duplicate
3. Check No. 39479 for \$690.00
4. Declaration and Power of Attorney
5. Specification with three sheets of drawings
6. Certificate of EXPRESS MAIL.
7. Return Post Card.

DATE SENT: April 17, 2000

WJR:imb

002140-66905560

"Express Mail" mailing label number

EL 388 803 025 US

Date of deposit: April 17, 2000

I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to the Commissioner of Patents and Trademarks, Washington, D.C. 20231.

Mattie M. Phillips

(Typed or printed name of person mailing paper or fee)

Mattie M. Phillips

(Signature of person mailing paper or fee)

C1118.00/C

Title: Evaporation of Liquids

Field of invention

This invention concerns evaporation of liquids.

Background to the invention

Directing a stream of air or an inert gas such as nitrogen onto the surface of a volatile solvent, is a technique used for evaporating such volatile liquids, but it suffers from the disadvantage that large volumes of inert gas or air, contaminated with vapour, must be disposed of. This can be expensive if the liquid is inflammable or toxic.

In one arrangement illustrated in Figure 1 of the accompanying drawings an inert gas, which for convenience will be generally referred to herein as nitrogen, is blown onto the surface of the solvent in one or more tubes. The tubes are usually located in a housing which is not hermetically sealed and the spent nitrogen is ducted to an area, such as a fume hood, so that the nitrogen contaminated with solvent vapour is led away in a manner which is considered safe.

As mentioned, this technique uses considerable quantities of nitrogen and generates large volumes of contaminated nitrogen which can be difficult to dispose of.

It is an object of the present invention to provide an improved method for exposing volatile liquids to an inert gas during an evaporation process, which reduces the problem identified above.

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Summary of the Invention

According to one aspect of the present invention there is provided a method of evaporating a liquid sample including a volatile solvent, comprising the step of continuously removing inert gas which is rich in solvent vapour from near the surface of the liquid.

This allows much easier collection of any toxic or inflammable material.

The gas may be withdrawn by a suction tube which is preferably maintained close to the surface of the liquid, as the liquid level drops due to evaporation.

Advantageously the evaporation rate may be increased by imparting an orbital motion to the container holding the liquid sample, causing the latter to spin around in the form of a vortex.

Preferably a liquid sample is contained within a chamber, and the volume of permanent gas recirculated to the chamber is progressively reduced, thereby to assist evaporation.

The invention also extends to an apparatus for performing the aforesaid method.

According to another aspect of the invention there is provided an apparatus for effecting the evaporation of a volatile solvent, wherein an inert gas, which is held in a sealed environment connected to at least one container containing the solvent, is recirculated via a condensing or absorbing device for absorbing or extracting solvent vapour entrained in the recirculating inert gas.

Preferably the container holding the liquid sample is mounted in a vortex evaporator, known per se.

Other aspects of the invention are defined in the appended claims.

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Brief description of the invention

The invention will now be described, by way of example only, with reference to the accompanying drawings in which:-

Figure 1 is schematic view of a known device in which an inert gas, such as nitrogen, is blown down onto the surface of a liquid to be evaporated;

Figure 2 shows apparatus in accordance with the invention for evaporating a volatile solvent;

Figure 3 shows apparatus closely similar to Figure 2 but operating in a different manner; and

Figure 4 shows a modification of the apparatus of Figure 3.

Detailed description of the drawings.

In Figure 2 a liquid sample 3 in a solvent mixture is contained in a tube container 2 inside a sealed chamber 1.

A suction tube 5 is placed with one end 11 in the container 2 at a position close above the top surface of the sample 3. This tube passes through the chamber 1 through a sealing means 12, through a further sealing means 13, and finally into a sealed refrigerated condensing vessel 6 in which the solvent vapour is stripped from the vapour/inert gas mixture.

A pipe 8 connects the refrigerated condensing vessel 6 to a pump 7 which draws the purified inert gas (eg air or nitrogen) from the vessel 6 and recirculates it through a further pipe 9 back to the chamber 1 again through a sealing means 14. From here the gas passes

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into the top of the container 2, as shown at 10, mixing with solvent vapour 4 which has been evaporated and passing back into the tube 5.

A heater 16, mounted around the pipe 9 near its inlet to the chamber, enables the cooled gas to be heated to a suitable temperature.

A pressure control system 15 allows the pressure in the system to be held at any required level, by venting gas to atmosphere or admitting gas into the system from a suitable source, eg a gas cylinder via a pressure regulator.

In operation the pump reduces the pressure in the refrigerated vessel 6, which causes the vapour 4 to be drawn through the tube 5 into the vessel 6 together with some inert gas, as above described. The temperature of the vessel 6 is maintained at a low enough value to condense the vapour, and any permanent gas passes through the vessel into the pump 7. Gas extracted by the pump 7 could be discharged to the atmosphere because it has been stripped of undesired vapours in the refrigerated vessel 6 or, as in this illustration, may be recirculated to continue to evaporate the sample.

If the gas from pump 7 is discharged to atmosphere, fresh gas is introduced to the chamber 1 via the pressure control means 15.

Suction by the pump 7 can, and preferable does, result in the pressure within the chamber 1 dropping as the evaporation process continues, in that the volume of air or nitrogen introduced by the control means 15 is arranged to be less than the volume of air or nitrogen withdrawn by the pump 7.

The reduced pressure in the chamber 1 assists in the evaporation of the solvent in the container 2 in a manner known per se.

The efficiency of the process is enhanced if the tube 5 is progressively lowered as the liquid level drops due to evaporation and held a few millimetres above the sample. This can be achieved manually using a means for lowering the tube 5 or raising the sample 3.

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Automatic positioning can also be used, using for example optical means to sense the position of the top of the liquid sample and using a sensor system to keep the bottom of the suction tube a few millimetres above the level. Electrical proximity means could also be used to sense the level.

In the example shown, a single sample tube container is illustrated. The process can be applied equally to arrays of many tubes and blocks with numerous wells, for example microtitre plates.

Although not shown in Figure 2, the sample tube 2 may be subjected to an orbital motion without alteration of the lateral orientation of the tube, such that the liquid 3 inside the tube moves around the tube in the form of a vortex. Such an arrangement is known per se as a vortex evaporator. The resultant agitation of the liquid in the tube increases the evaporation rate, allowing a more rapid extraction of vapour-rich gas to take place through the suction tube 5.

In place of a refrigerated condensing vessel 6, there could be substituted any suitable absorbent material, such as molecular sieves, activated charcoal, silica gel etc. In this case the gas passing back into the vessel 6 will not have been cooled, so that the heater 16 may not be required.

Referring now to Figure 3, there is shown a modification of the apparatus of Figure 2 which operates in a different manner.

In this arrangement the same reference numerals are used as in Figure 2. However, the heater 16 is here disposed around the external part of the tube 5 between the sealed chamber 1 and the condensing vessel 6. Moreover, the end of the tube 5 does not extend to a position close above the surface of the sample, but is instead shaped in the form of a nozzle 17.

In this arrangement the flow of gases and vapours in the tube 5 and pipes 8 and 9 is opposite to that shown in Figure 2, the operation of the arrangement being as follows:

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The pump 7 sucks inert gas (typically nitrogen) mixed with solvent vapour out of the sealed container 1 through the pipe 9 and passes it through the refrigerator condenser 6 or other device for absorbing or extracting the solvent vapour. The purified gas is then ducted into the tube 5 which terminates in the nozzle 17. The resultant jet of gas passing over the liquid 3 in the tube 2 tends to pick up and remove the layer of saturated vapour 4 above the liquid. The contaminated nitrogen 10 then escapes from the tube container 2 and is sucked out of the chamber 1 by the action of the pump 7, as above mentioned.

A pressure control system 15 in the pipe 9 maintains the pressure in the system at the required level by adding or removing nitrogen to or from the system. The heater 16 is used to warm the gas emerging from the condenser 6. The tube 5 and pipe 9 are sealed at 12, 13 and 14 where they enter and leave the container 1 and the condenser 6.

Although, again, only a single sample tube container 2 is illustrated, the system can be applied equally to a chamber housing a plurality of tubes, usually arranged in an array, and is equally applicable to arrangement in which the containers are replaced by blocks containing a plurality of wells, for example microtitre plates.

The or each sample tube container 2 is preferably subjected to an orbital motion without alteration of the lateral orientation of the tube, such that the liquid 3 inside the tube moves around the tube in the form of a vortex, such an arrangement being known as a vortex evaporator.

As with the apparatus of Figure 2, the condenser 6 could be replaced by a suitable absorbent material, eg molecular sieves etc, in which case the heater 16 may again not be required.

Referring now to Figure 4, there is shown a further modification of the apparatus of Figure 3, in which the same reference numerals are used for similar parts.

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Here there are three containers 2 containing liquid samples 3, only the left-hand one being referenced. The top of each container is secured to a manifold 20 by gas-tight seals 22. The manifold is divided into upper and lower compartments 24, 26, the lower of which has apertures 28 extending into the outer regions of each container 2. The upper compartment 24 is similarly provided with a passageway 30 for each container which passes through the lower compartment from the centre at the top of the container.

The lower compartment 26 is connected by the pipework 5 to the heater 16, while the upper compartment chamber 24 is connected by the pipework 9 to a pump, such as the pump 7 shown in Figures 2 and 3, which in turn is connected to the condenser 6.

The operation of the modified apparatus is similar to that for Figure 3. Thus inert gas mixed with solvent vapour is drawn through the passageways 30 and the upper compartment 24 by the pump 7 and is conveyed from there to the condenser 6. At the same time purified gas, warmed by the heater 16, is ducted into the lower compartment 26, and from thence through the apertures 28 and into each container 2, so as to purge the saturated vapour above each liquid sample and enable it to be extracted through the passageways 30.

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CLAIMS

1. A method of evaporating a liquid sample including a volatile solvent, comprising the step of continuously removing inert gas which is rich in solvent vapour from near the surface of the liquid.
2. A method according to claim 1 in which the gas is cooled in a condenser wherein the vapour condenses, allowing the purified gas to be recirculated.
3. A method according to claim 1 or claim 2 in which the evaporation rate is increased by imparting an orbital motion to the container holding the liquid sample, causing the latter to spin around in the form of a vortex.
4. A method according to any one of claims 1 to 3 in which the liquid sample is contained within a chamber, and the volume of gas recirculated to the chamber is progressively reduced, thereby to assist evaporation.
5. Apparatus for performing the method according to any one preceding claim in which the liquid is held in a container, and said gas is withdrawn by a suction tube whose open end is maintained close above the top surface of the liquid as the liquid level drops in the container due to evaporation.
6. Apparatus according to claim 5 as dependent upon claim 2 in which the tube is connected to a refrigerated condensing device through which the permanent gas is passed by a pump for recirculation.
7. Apparatus according to claim 5 or claim 6 in which the container is mounted in a vortex evaporator.

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8. Apparatus according to any one of claims 5 to 7 further comprising a vacuum chamber in which the container is mounted and means for progressively reducing the volume of gas which is recirculated to the container by the pump.
9. Apparatus for effecting the evaporation of a volatile solvent, wherein an inert gas, which is held in a sealed environment connected to at least one container containing the solvent, is recirculated via a condensing or absorbing device for absorbing or extracting solvent vapour entrained in the recirculating inert gas.
10. Apparatus according to claim 9 in which the inert gas is returned directly into the upper end of the container.
11. Apparatus according to claim 10 in which the inert gas is directed into the container through nozzle means which directs the gas directly onto the surface of the solvent in the container.
12. Apparatus according to any one of claims 9 to 11 in which a pressure control means introduces or extracts inert gas, in order to maintain a predetermined pressure in the recirculating system.
13. Apparatus according to any one of claims 9 to 12 in which the inert gas is recirculated by means of a pump.
14. Apparatus according to any one of claims 9 to 13 in which a gas reservoir means is provided in which inert gas is stored under pressure for reuse as required, with pressure controlling means limiting the pressure of the gas leaving the reservoir to a desired level.
15. Apparatus according to any one of claims 9 to 14 in which the container is mounted in a vortex evaporator.
16. Apparatus according to any one of claims 5 to 15 in which the inert gas is nitrogen.

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17. Apparatus according to any one of claims 5 to 16 in which a heater is provided in the recirculating system downstream of the condensing device.

18. Apparatus according to any one of claims 9 to 15 in which the container is open-topped for accommodation in the sealed environment.

19. Apparatus according to any one of claims 9 to 15 in which a plurality of containers are connected to a manifold through which the inert gas is recirculated.

20. Apparatus according to claim 19 in which the manifold is divided into two compartments, one being connected to a pump for extracting the mixture of the inert gas and solvent vapour and the other feeding recirculated inert gas from the condensing or absorbing device.

21. A method of, or apparatus for, evaporating a liquid sample substantially as herein described with reference to, and as shown in, Figures 2 to 4 of the accompanying drawings.

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AbstractTitle: Evaporation of Liquids

A liquid sample 3 held in an open container 2 and including a volatile solvent, is evaporated by continuously removing inert gas rich in vapour by means of a suction tube 5 whose open end is held close to the top surface of the liquid. Vapour in the gas is then extracted in a refrigerated condensing vessel 6, from whence the purified gas is fed by a pump 7 for recirculation back into a sealed chamber 1 in which the container is mounted. In a modification, the inert gas is fed directly through a nozzle 17 into the container 2 while the gas/vapour mixture is extracted from the chamber 1 for recirculation, after passing through a condenser 6 or the like.

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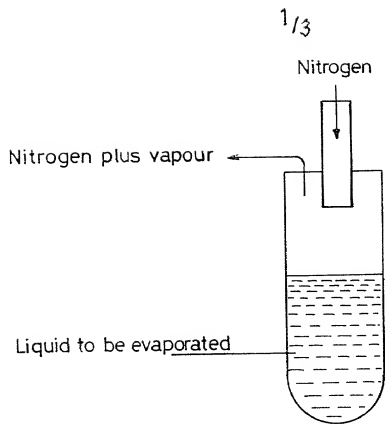


Fig. 1
(PRIOR ART)

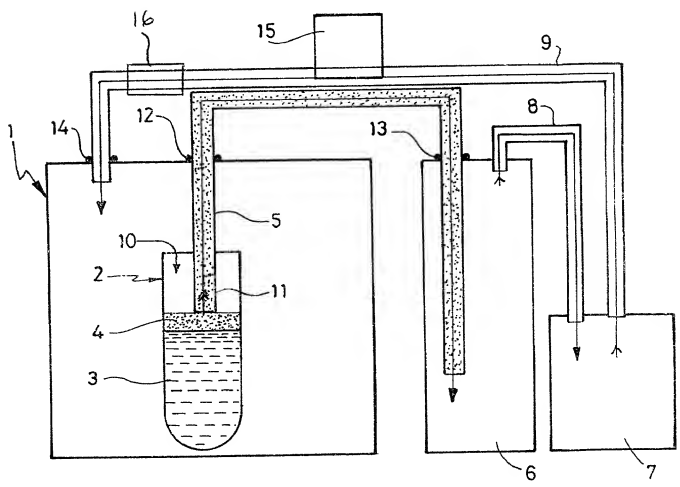


Fig. 2

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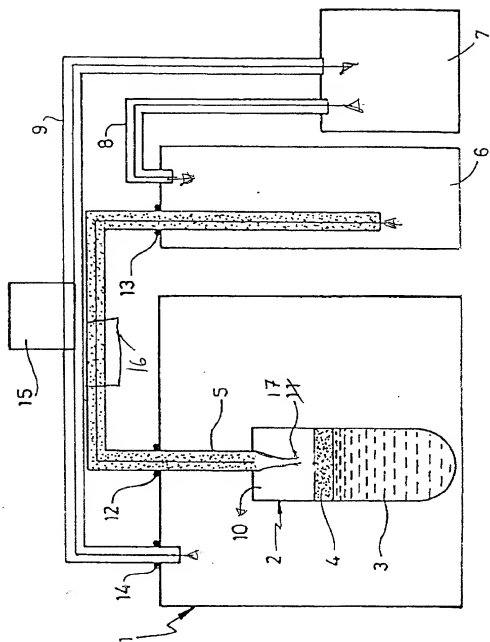
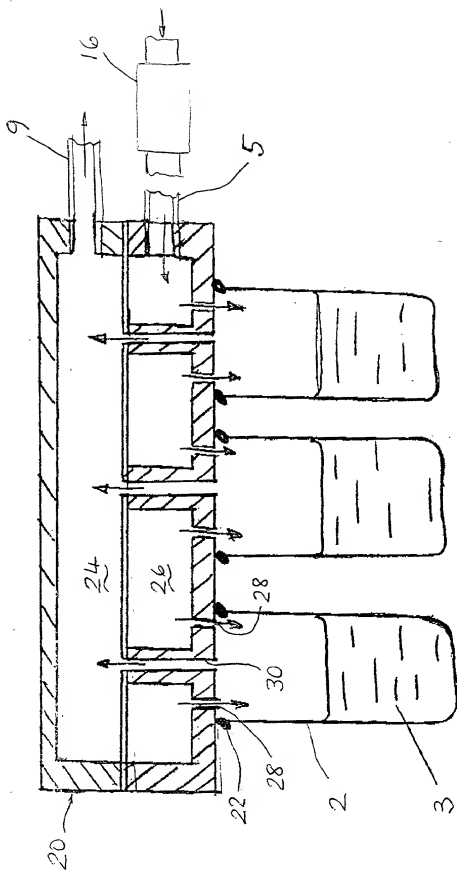


Fig. 3

FIG. 4

DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated
below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original,
first and joint inventor (if plural names are listed below) of the subject matter which is claimed and
for which a patent is sought on the invention entitled Evaporation of Liquids, the specification of
which:

 X is attached hereto.

 was filed on as

PCT Application Serial No.

and was amended on (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification,
including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this
application in accordance with Title 37, Code of Federal Regulations, Section 1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119 of any
foreign application(s) for patent or inventor's certificate listed below and have also identified below
any foreign application for patent or inventor's certificate having a filing date before that of the
application on which priority is claimed:

0690

I hereby claim the benefit under Title 35, United States Code Section 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, Section 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, Section 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application.

And I hereby appoint Wm. Marshall Lee, Registration No. 16,853, John M. Mann, Registration No. 17,775, Thomas E. Smith, Registration No. 18,243, Dennis M. McWilliams, Registration No. 25,195, James R. Sweeney, Registration No. 18,721, William M. Lee, Jr., Registration No. 26,935, Glenn W. Ohlson, Registration No. 28,455, David C. Brezina, Registration No. 34,128, Jeffrey R. Gray, Registration No. 33,391, Timothy J. Engling, Registration No. 39,970, Gregory B. Beggs, Registration No. 19,286, Gerald S. Geren, Registration No. 24,528 and Peter J. Shakula, Registration No. 40,808 as my attorneys to prosecute this application and to transact all business in the Patent and Trademark Office connected herewith. It is requested that all communications be directed to Lee, Mann, Smith, McWilliams, Sweeney & Ohlson, P.O. Box 2786, Chicago, Illinois 60690-2786, telephone number (312) 368-1300.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first inventor: Michael COLE

Signature [Signature] Date 7/1/00

Country of Residence: United Kingdom

Country of Citizenship: United Kingdom

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